IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:	:	**PATENT APPLICATION**
	:	
Stephen Moreton	:	Silica-Based Indicating Desiccants
	:	
Serial No.: 10/549,670	:	Group Art Unit: 1797
	:	
Filed: July 3, 2006	:	Examiner: B. Kilpatrick

DECLARATION OF STEPHEN MORETON, Ph.D.
UNDER 37 C.F.R. § 1.132

1. I, Dr. Stephen Moreton, am the inventor of the "Silica-Based Indicating Desiccants" that are the subject of U.S. Patent Application No. 10/549,670 (the " '670 application"). In 1989 I received my Ph.D. in inorganic chemistry from Edinburgh University.

2. I have read and am familiar with the '670 application.

3. I have generated data to demonstrate the effectiveness of common iron salts in conjunction with a source of bromide, as humidity indicators at RH from 0 to 20% in silica gel desiccant.

4. Silica gels were prepared containing different iron salts together with sodium bromide using the method of example 4 in the '670 application as filed. These impregnated gels were dried for around 4 hours and exposed to air streams at 10, 20, 40 & 80 % relative humidity for 7 hours, as described in the patent. The colours of the gel were noted, and measured using a Chromameter, according to the patent method. The samples were also photographed and the photographs are appended to this Declaration as Exhibits 1-6. Details, and the results, for various iron salts are described below.

5. The humidified silica gel (2.5 – 6.0 mm diameter granules) contained approximately 23 % water (as determined by weight loss at 145 °C). For all of the examples below the

quantities of gel, and of iron and bromide salts, were calculated to give 0.24 % Fe and 0.44 % Br in the dried product, with a Br:Fe weight ratio of 1.83. This works out at 0.57 g NaBr in each case (when Br is present), and about 126 – 128 grams of humidified gel. For the nitrate, the level of bromide was doubled to give Br:Fe of 3.7 for reasons explained below.

6. Potassium iron(III) sulphate, $KFe(SO_4)_2$

This is the potassium analogue of the ammonium iron(III) sulphate, or iron alum, employed in the patent application. A solution was prepared containing 1.4275 g of this in 10 ml water. This served as the control. Another solution contained the same plus 0.5665 g sodium bromide.

7. The solutions were mixed with 128.0 & 127.3 grams of humidified silica gel respectively. After drying at 145 °C the gels were analysed, and tested as described above. Results are shown in Table 1.

Table 1. Indicating behaviour of the dried potassium iron alum gel.

Composition	% R.H.	L*	a*	b*	Colour
No Br (control)	0	46.69	+5.22	+26.51	Light orange
	10	48.54	+3.32	+30.00	Light orange
	20	50.02	+2.30	+24.98	Light orange
	40	53.63	+2.34	+16.38	Pale yellowish orange
	80	58.97	-0.39	+9.15	Pale yellowish
With Br	0	36.77	+14.15	+34.20	Amber/brown
	10	47.63	+3.74	+30.96	Light orange
	20	51.01	+2.62	+27.60	Light orange
	40	51.25	+2.39	+19.98	Pale yellowish orange
	80	59.27	-0.44	+10.54	Pale yellowish

8. It is clear from the data in Table 1 and the photographs in Exhibit 1 that the presence of the Br source gives a major improvement in colour change for the indicating desiccant between 0 and 20% RH compared to the control sample without a source of Br.

9. Ammonium iron(II) sulphate, $(NH_4)_2Fe(SO_4)_2.6H_2O$

This compound, also known as ferrous ammonium sulphate, or Mohr's salt, will react with atmospheric oxygen and oxidise to the iron(III) state during the drying stage, and so behave similarly to ammonium iron(III) sulphate, or iron alum. A solution of 1.6854 g of this in 10 ml of water was prepared (the control) and another containing the same plus 0.5665 g sodium bromide. The solutions were mixed with humidified gel and dried at 145°C and tested as above. Results are shown in Table 2.

Table 2. Properties of the dried Mohr's salt gel.

Composition	% R.H.	L*	a*	b*	Colour
No Br (control)	0	42.94	+10.70	+26.65	Light orange
	10	46.02	+10.42	+28.34	Light orange
	20	42.41	+12.28	+28.33	Light orange
	40	52.68	+3.22	+15.59	Pale orange
	80	57.47	+1.20	+14.26	Pale yellowish
With Br	0	40.18	+7.55	+31.77	Yellow/light brown
	10	40.94	+9.83	+24.44	Light amber
	20	44.40	+10.53	+28.28	Light orange
	40	51.88	+6.47	+24.99	Pale orange
	80	54.75	+1.26	+13.02	Pale yellowish

10. Again, the data in the table above, and the photographic results in Exhibit 2, show that the presence of a source of Br along with the iron(II) double salt gives a significant improvement in colour change between 0 and 20% RH.

11. Iron(III) sulphate, $Fe_2(SO_4)_2.5H_2O$ (Ferric sulphate)

A solution of 1.0530 g of this in 10 ml of water was prepared (the control) and another containing the same plus 0.5665 g sodium bromide. Each solution was mixed with humidified gel, dried at 145°C and tested as above. Results are shown in Table 3.

Table 3. Properties of the dried ferric sulphate gel.

Composition	% R.H.	L*	a*	b*	Colour
No Br (control)	0	46.49	+3.68	+28.84	Light orange
	10	49.62	+0.72	+24.38	Light yellow
	20	53.90	+0.72	+20.08	Light yellow
	40	56.12	-0.02	+13.30	Pale yellow
	80	57.28	-1.15	+6.48	Almost colourless
With Br	0	31.32	+18.26	+26.76	Deep amber/brown
	10	45.69	+5.19	+32.57	Light orange
	20	49.77	+3.54	+28.18	Light yellow
	40	54.55	+1.49	+20.71	Light yellow
	80	59.21	-1.69	+9.90	Pale yellow

12. The data in the table, and the photographic results shown in Exhibit 3, each show a marked increase in the colour change for the indicating desiccant between 0 and 20% RH.

13. Iron(III) chloride, $FeCl_3.6H_2O$ (Ferric chloride)

A solution of 1.1617 g of this in 10 ml of water was prepared (the control) and another containing the same plus 0.5665 g sodium bromide. Each solution was mixed with humidified gel and dried at 100°C for 3 hours, before being tested as above. The gentler drying condition was selected to reduce the tendency of the chloride to undergo hydrolytic decomposition.

Table 4. Properties of the dried ferric chloride gel.

Composition	% R.H.	L*	a*	b*	Colour
No Br (control)	0	49.20	-1.22	+26.98	Yellow
	10	49.77	-1.44	+31.13	Yellow
	20	53.64	-2.23	+26.54	Yellow
	40	58.91	-3.15	+15.92	Pale yellow
	80	59.49	-2.79	+8.69	Pale yellow
With Br	0	34.73	+14.92	+33.48	Deep amber/brown
	10	38.30	+10.70	+35.12	Amber
	20	45.71	+6.31	+33.79	Light orange
	40	54.85	-1.62	+24.90	Light yellow
	80	58.13	-3.28	+22.38	Light yellow

14. The data in the table, and the photographic results shown in Exhibit 4, each show a marked increase in the colour change for the indicating desiccant between 0 and 20% RH.

15. Iron(III) nitrate, $Fe(NO_3)_3.9H_2O$

Ferric nitrate was used with a lower drying temperature (80°C) and twice as much bromide than for the other salts. A solution of 1.7366 g of this in 10 ml of water was prepared (the control) and another containing the same plus 1.1330 g sodium bromide (i.e. Br:Fe weight ratio 3.7). Each solution was mixed with humidified gel and dried at 80 °C for 3 hours, before being tested as above. The gentler drying conditions, and higher bromide level, were selected to reduce the tendency of the nitrate to undergo hydrolytic decomposition. Results are shown in Table 5.

Table 5. Properties of the ferric nitrate gel dried at 80 °C.

Composition	% R.H.	L*	a*	b*	Colour
No Br (control)	0	53.49	+0.36	+17.41	Light yellow
	10	50.83	+0.68	+18.70	Light yellow
	20	55.92	-0.25	+12.92	Pale yellow
	40	59.72	-0.39	+9.26	Pale yellow, almost colourless
	80	59.94	-0.39	+6.16	Almost colourless
With Br	0	45.36	+6.71	+33.11	Amber
	10	42.86	+9.15	+35.34	Amber
	20	48.25	+3.83	+28.40	Light orange
	40	54.32	+1.44	+22.62	Light yellow
	80	55.96	-0.42	+14.61	Pale yellow

16. The data in the table, and the photographic results shown in Exhibit 5, both show a marked increase in the colour change for the indicating desiccant between 0 and 20% RH, compared to the colour change for the desiccant without the Br source.

17. Iron(II) sulphate, $FeSO_4.7H_2O$ (Ferrous sulphate)

A solution of 1.1946 g of this in 10 ml of water was prepared (the control) and another containing the same plus 0.5665 g sodium bromide. Each solution was mixed with humidified gel and dried and tested as above. Results are shown in Table 6.

Table 6. Properties of the dried iron(II) sulphate gel.

Composition	% R.H.	L*	a*	b*	Colour
No Br (control)	0	41.92	+7.76	+24.72	Amber
	10	46.54	+8.06	+28.46	Orange
	20	49.25	+6.58	+24.34	Orange
	40	52.71	+3.85	+20.75	Light orange & colourless, uneven
	80	57.96	+1.48	+14.96	Pale yellow & colourless, uneven
With Br	0	34.90	+10.18	+31.31	Deep amber
	10	45.15	+10.58	+32.81	Amber
	20	47.85	+8.55	+30.74	Orange
	40	52.05	+3.72	+19.93	Light orange & colourless, uneven
	80	58.70	+0.46	+12.87	Pale yellow & colourless, uneven

18. The data in the table, and the photographic results shown in Exhibit 6, each show a marked increase in the colour change for the indicating desiccant between 0 and 20% RH, compared to the colour change for the desiccant without the Br source.

I declare that the foregoing is true and correct, that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: September 1, 2009

S. Moreton

Stephen Moreton, Ph.D.

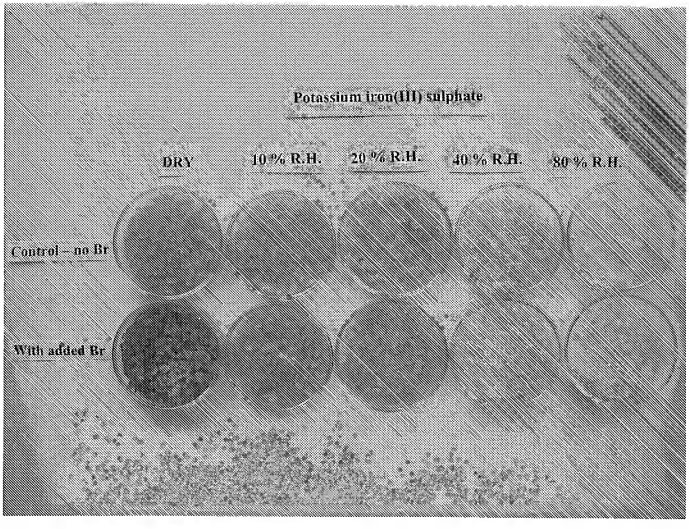
Potassium iron(III) sulphate
DRY
10 % R.H.
20 % R.H.
40 % R.H.
80 % R.H.
Control – no Br
With added Br

Exhibit 1

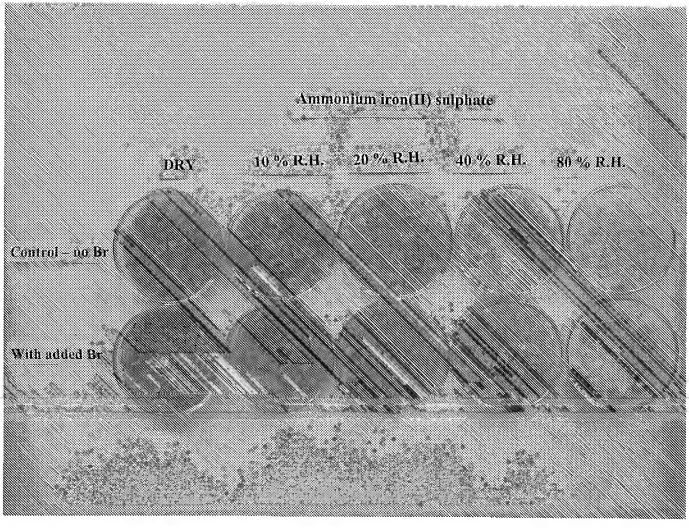
Ammonium iron(II) sulphate
DRV
10 % R.H.
20 % R.H.
40 % R.H.
80 % R.H.
Control – no Br
With added Br

Exhibit 2

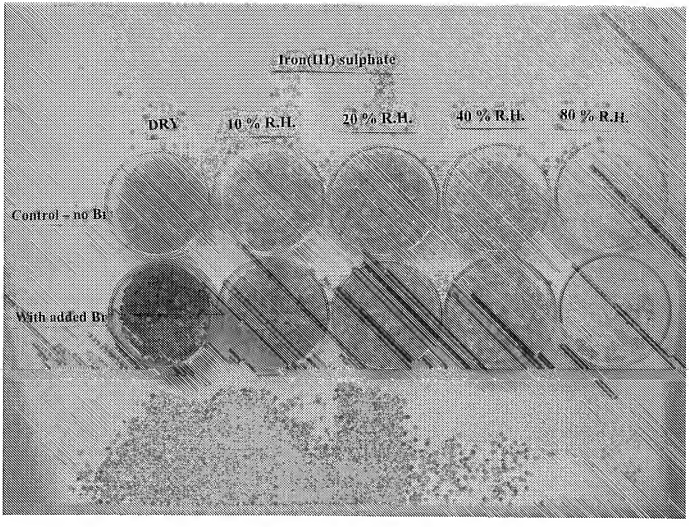
Iron(II) sulphate
DRY
10 % R.H.
20 % R.H.
40 % R.H.
80 % R.H.
Control – no Bi
With added Bi

Exhibit 3

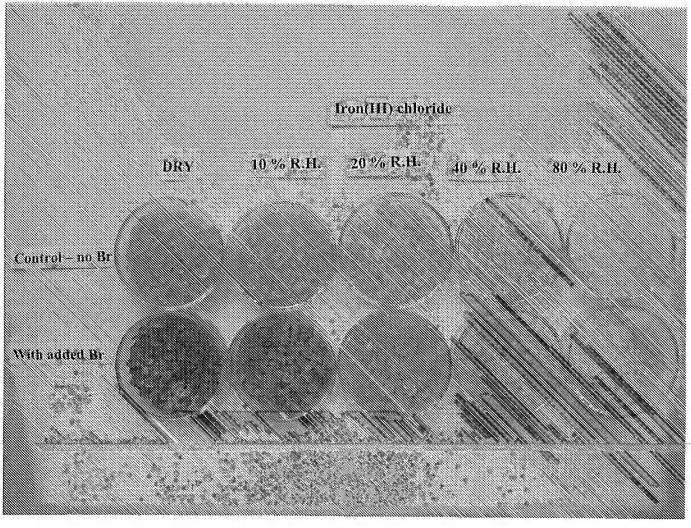
Iron(III) chloride
DRY
10 % R.H.
20 % R.H.
40 % R.H.
80 % R.H.
Control – no Br
With added Br

Exhibit 4

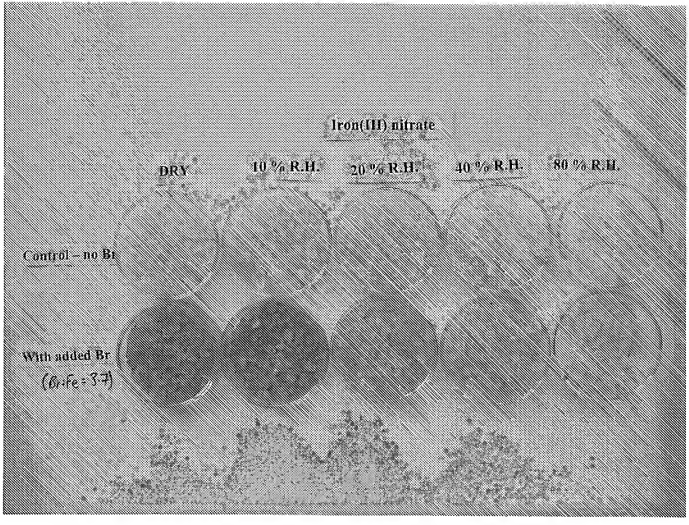
Iron(III) nitrate
DRY
10 % R.H.
20 % R.H.
40 % R.H.
80 % R.H.
Control – no Br
With added Br
(6:1 Fe : Br)

Exhibit 5

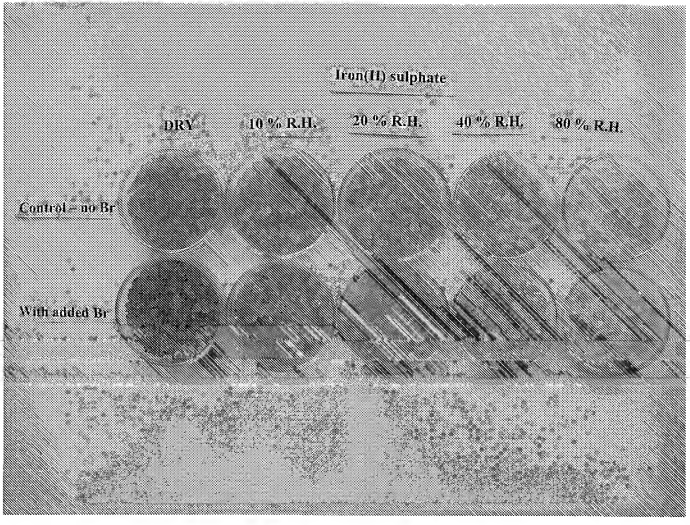
Iron(II) sulphate
DRY
10 % R.H.
20 % R.H.
40 % R.H.
80 % R.H.
Control – no Br
With added Br

Exhibit 6